HANDBOOK FOR THE DEPARTMENT OF ENERGY'S MIXED ANALYTE PERFORMANCE EVALUATION PROGRAM (MAPEP)

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I. INTRODUCTION

Compliance and quality assurance issues associated with DOE regulatory authorities typically require analytical services under contract with DOE to participate in a variety of performance evaluation programs (PEPs). The primary objective of the PEPs is to foster reliability and credibility for the analytical results used in the decision making process, particularly as it relates to the environment and public health and safety. Each PEP checks for specific analytical proficiencies in radiological, stable inorganic, or organic analyses. Regulatory requirements, however, frequently include analyses for radiological and nonradiological constituents of the same sample. A PEP for quantifying these mixed analytes was not previously available. The Analytical Services Division of EM established the MAPEP to address this deficiency and to help assure the quality of analytical services across the DOE Complex.

The Radiological and Environmental Sciences Laboratory (RESL), under the direction of DOE-HQ Environmental Safety and Health (ES&H) will administer the MAPEP. MAPEP samples will include water, soil, air filter and vegetation matrices with environmentally important stable inorganic, organic, and radioactive constituents. Consolidating the major analytes of interest into a single PEP will provide a more representative mixed analyte sample and an efficient means for laboratories to demonstrate the required proficiencies.

II. PARTICIPATION

All laboratories that perform environmental analytical measurements for EM (i.e., radiological, stable inorganic, and/or organic analyses, solely or in any combination) are required to participate in the MAPEP (Memorandum from the Assistant Secretary for Environmental Management, May 31, 1994, Newberry:3-7615). MAPEP samples are a mixed **analyte** matrix, NOT a mixed **waste**: "MAPEP samples are analytical standards or a product generated for the purpose of securing and evaluating analytical services; they are not hazardous waste and they are not samples of hazardous waste... Thus, a laboratory participating in the MAPEP is in the process of establishing its eligibility and credentials to do DOE analytical work." (Memorandum OCC-95-189, Office of the Chief Council, October 16, 1995). Participation is defined as requesting the performance sample materials, completing the appropriate analyses, reporting the results to RESL, and implementing any corrective actions. Participation may be requested by writing the MAPEP Coordinator:

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e-mail: MARLETGM@ID.DOE.GOV

A request for participation should include a shipping (do not use a post office box number) and correspondence address, a contact person for each, appropriate phone numbers, FAX number, e-mail address, any special shipping instructions, the current NRC or state license number for the laboratory or a statement of NRC license exemption, and the license or exemption expiration date. Since the MAPEP samples have a radioactive component, an NRC license or exemption is required by all receiving laboratories. Exemptions should specify the DOE contract number for the laboratory.

Participating laboratories are required to have appropriate radiological control measures and a QA/QC plan. Guidance for the QA/QC plan can be found in *Analytical Laboratory Quality Assurance Guidance in Support of EM Environmental Sampling and Analytical Activities (DOE/EM-0159P, May 1994).* Furthermore, in performing sample analyses the participating laboratory accepts title and ownership of the MAPEP sample and becomes the generator of any resulting waste or sample residues.

III. SAMPLE PREPARATION AND CHARACTERIZATION

Liquid MAPEP samples are prepared from radiological and stable inorganic standards traceable to the National Institute of Standards and Technology (NIST). Final concentrations for these analytes are calculated from the certified standard value and the appropriate dilution. A known quantity of standard is combined and diluted to a known final volume with 2-5% (v/v) nitric acid and appropriately characterized water. The nitric acid is high-quality double distilled with certified concentrations for known contaminants. Organic analytes are added to a separate non-acidic sample with radiological and stable inorganic components. All sample containers are acid-washed polyethylene or other suitable material. Custody seals are applied over closures and the sample bottles are stored in a secured facility until distribution.

Solid samples are prepared from natural matrices fortified with NIST traceable standards as needed for various analytes of interest. The sample is characterized, homogeneity is assessed and target analyte concentrations are verified prior to sample distribution. Known values for the analytes, whenever possible, are calculated from the certified standard values and the appropriate dilution. If this method is not feasible, the known values are derived from the sample characterizations. Sample handling and storage procedures are similar to those for the liquid sample.

MAPEP samples are typically not classified as radioactive (total activity < 2 nCi/gram) by the Department of Transportation (DOT). Other hazardous components are typically below the Resource Conservation and Recovery Act (RCRA) regulatory concentrations. Sample descriptions delineating the major analytes of interest, concentration ranges, etc., will be provided. Each participant is responsible for reviewing their own compliance requirements and must determine if the analytical procedures utilized result in a mixed waste. Analysis shall not proceed without full compliance to all appropriate regulatory authorities.

IV. SAMPLE DISTRIBUTION

Samples are distributed semiannually. A sample description will be available on the Internet to sample distribution (see Appendix B for a typical sample description). The MAPEP Coordinator must be notified of any special shipping requirements or other problems pertaining to the sample at this time. The participants must ensure that they are authorized to receive a mixed analyte sample and that their standard operating procedures incorporate appropriate sample management and waste disposal practices. Acceptance of the sample means that the participating laboratory takes title and ownership of the sample. Excess sample or associated residues cannot be returned to RESL. Sample analysis shall not be initiated if approved treatment, storage, or disposal options are not available.

V. SAMPLE ANALYSES

Analyses are required for only those analytes that are a component of the participant's routine EM analytical workload or compliance requirements (i.e., a complete sample analysis may not be appropriate). Laboratories are, however, expected to report results for a targeted analyte if the determination is typically given by the analytical methodology utilized (e.g., if Pu-238 and Pu-239 are targeted analytes, and results for Pu-239 are reported utilizing Alpha Spectrometry, results for Pu-238 are also expected to be reported). The receiving laboratory can split the sample and subcontract analyses if this is routinely performed. If a subcontracted laboratory is already participating in the MAPEP, a sample split is not required. The subcontracting laboratory can simply request (use the Additional Addresses option in the MAPEP data entry program) that a copy of their MAPEP performance report be sent to the contracting laboratory. This will document MAPEP participation for all interested parties and will also prevent redundant analyses. The same analytical procedures employed for routine analyses should be utilized for MAPEP samples. Participants are typically allowed 60-90 calendar days to complete those analyses not controlled by regulatory holding times. The deadline for reporting results will be specified for each sample distribution.

Although analytical methods are not prescribed by MAPEP, standard analytical procedures will be utilized to independently characterize the MAPEP samples. These analytical techniques include alpha spectrometry, beta counting, gamma spectrometry, inductively coupled plasma (ICP) atomic emission spectroscopy, ICP mass spectrometry, gas chromatography, gas chromatography/mass spectrometry, etc.
Activities for radiological analytes are typically sufficient to provide less than 10% counting uncertainty with reasonable sample size and count time. Similar uncertainties should be achievable for most stable inorganic/organic analytes. The amount of sample is, however, limited. Therefore, the activity and concentration ranges indicated in the sample description should be used to select the optimum quantity of sample for each analysis.

VI. REPORTING RESULTS

Analytical results are reported to RESL via the Internet. Data entry and edit screens are available for reporting the analytical results, and a hard copy record can be printed for laboratory records and/or review. The program guides the user through selection of Method Codes for radiological (see Appendix C), stable inorganic (see Appendix D), and organic (see Appendix E) analyses. Data are entered directly into the MAPEP database via the Internet. Specific instructions for using the data entry program are provided in Appendix F.

The MAPEP program will NOT accept any hard copy, floppy disks or other electronic media without prior authorization. A laboratory's performance report can be mailed to additional addresses if desired. Participants should strive to keep their respective address and contact information current.

Participants are required to report only ONE result for each appropriate analyte. Each reported radiological result must be accompanied by an estimate of its uncertainty in the units of measurement (NOT as a percent), and both numbers should follow the rules for significant figures. The MAPEP strongly encourages that all results (including stable inorganic and organic analyses) be reported with uncertainty estimates. If the reported result is actually a mean of several replicate analyses, the reported uncertainty should also be the MEAN of the INDIVIDUAL uncertainties at one standard

deviation. Do NOT combine the variances associated with the individual uncertainties for replicate measurements, even though this should typically be performed. The larger individual uncertainties associated with a single analysis are of interest to MAPEP since they are more indicative of routine performance. For example, assume three replicate analyses provided the following results and individual uncertainties: 101 +/- 12, 108 +/- 15, 110 +/- 16. The mean result is (101+108+110)/3=106 and the MEAN INDIVIDUAL UNCERTAINTY is (12+15+16)/3=14. The result and total uncertainty as reported for MAPEP is 106 +/- 14. The total uncertainty should be reported at the one sigma level.

The uncertainty characterizes the range about the result within which the true value is expected to lie (result +/- uncertainty). The uncertainty provides a probabilistic statement about the extent to which the result may be inaccurate. Because of Poisson counting statistics, a unique uncertainty can be propagated for each radiological result. This is not typically the case for stable element analyses where average uncertainties are assigned for different analytes and concentration ranges. The exact method for estimating the uncertainty is not prescribed here since the reported uncertainty for MAPEP analyses should reflect the actual methods used for data generated on routine real-world samples. For guidance, however, it is preferred to estimate all uncertainty components, including those derived from a complete statistical analysis (Type A, s_A) and those evaluated by other means (Type B, s_B), as approximations to standard deviations. This convention follows that proposed by the Bureau International des Poids et Mesures (BIPM) and as suggested in several standard references (NIST Technical Note 1297, 1993; ISO/IEC/OIML/BIPM Expression of Uncertainty: 1993 (E); NCSL Information Manual - Determining and Reporting Measurement Uncertainties, RP-12, 1994; ANSI N42.14-1991; NCRP Report No. 58, second edition, 1985). It allows all of the uncertainty components to be propagated into a total combined uncertainty by statistical rules and the combination of variances:

$$\mu = \sqrt{s_A^2 + s_B^2}$$

where μ = the combined uncertainty and the other variables are as described above.

For example, let R = the analytical result, ΔR = the total combined uncertainty in the result, U1 = an uncertainty component involved in the calculation of the result (such as a pipette calibration), $\Delta U1$ the uncertainty in the pipette calibration (derived statistically as the standard deviation of 10 measurements, i.e., an example of Type A uncertainty), U2 = a second uncertainty component (such as the value of a calibration standard used in calculating the result), $\Delta U2$ = the uncertainty of the calibration standard (derived from a standard certificate at one standard deviation, i.e., an example of Type B uncertainty), U3 = a third uncertainty component (such as a weight measurement), $\Delta U3$ the uncertainty in the weight measurement, U4 = a fourth uncertainty component (such as a volume measurement), $\Delta U4$ = the uncertainty in the volume measurement, etc. Note that all uncertainty components, including Type B uncertainty, should be estimated at one standard deviation. The equation used to calculate the total combined uncertainty in the result is given by:

$$\Delta R = R * \sqrt{\left[\frac{\Delta U1}{U1}\right]^2 + \left[\frac{\Delta U2}{U2}\right]^2 + \left[\frac{\Delta U3}{U3}\right]^2 + \left[\frac{\Delta U4}{U4}\right]^2 + \ldots.}$$

This example is for illustrative purposes only; frequently the uncertainty components cannot be derived directly but must rely on the mathematical manipulation of other measurable quantities. In this event, the specific error propagation formulas for the various mathematical functions (i.e., addition, subtraction, multiplication, division, exponential, etc.) must be utilized. These formulas and a detailed discussion on error propagation can be found in the references cited above and other statistical and analytical references. A statistician may also be consulted. The uncertainty for MAPEP analyses should be reported at one standard deviation.

When entering inorganic and organic analytical results, the uncertainty field associated with the result is optional for input. If the laboratory propagates uncertainties for the analytes being reported, then the uncertainty field must be used to record the uncertainty result for the inorganic or organic analyte. It is important to report all uncertainties at one standard deviation in the units of measurement, NOT in percent. Many MAPEP participants utilize EPA methodology and therefore do not routinely report uncertainties. The MAPEP, however, stresses the importance of determining the uncertainty of a measurement as outlined in the ISO, NIST, and other references cited above.

VII. EVALUATION

Acceptance criteria were developed from a review of precision and accuracy data compiled by other PEPs, the analytical methods literature, from several MAPEP pilot studies, and from what is considered reasonable, acceptable, and achievable for routine analyses among the more experienced laboratories. The acceptance criteria are designed to be pragmatic in approach and may be changed as warranted.

For each reported radiological and inorganic analyte, the laboratory result and the RESL reference value will be used to calculate a relative bias:

$$\%\ BIAS = \frac{(100)(LABORATORY\ RESULT - RESL\ REFERENCE\ VALUE)}{RESL\ REFERENCE\ VALUE}$$

For each reported organic analyte, the laboratory result, the mean of all reported results and the standard deviation of all results (less outliers) will be used to calculate a Z-score:

$$Z - Score = \frac{(100)(LABORATORY\ RESULT - MEAN\ OF\ ALL\ DATA)}{STANDARD\ DEVIATION\ OF\ ALL\ DATA}$$

The relative bias will place the laboratory result in one of three categories:

1) ACCEPTABLE..... BIAS <= 20%
2) ACCEPTABLE WITH WARNING…. 20% < BIAS <= 30%
3) NOT ACCEPTABLE..... BIAS > 30%

The Z-Score will place the laboratory result in one of three categories:

1) ACCEPTABLE..... Z-Score <= 2.0
2) ACCEPTABLE WITH WARNING…. 2.0 < Z-Score <= 3.0
3) NOT ACCEPTABLE..... Z-Score > 3.0

The reported uncertainty is not currently used as part of the acceptance criteria, but it will be used to flag a potential area of concern. Activity levels and other analyte concentrations for MAPEP samples are typically sufficient to permit analyses with uncertainties of 10% or less, but it is unreasonable to expect the uncertainty for a single analysis of a routine sample to be much lower than the 10% value. Variations in counting efficiencies, chemical yields, analytical methods, sample size, count times, difficult analyses, etc., will likely cause some uncertainties to exceed the 10% value. A meaningful routine analysis, however, will not over inflate the uncertainty estimate. The MAPEP will provide some feedback to the participants regarding the uncertainties reported with their results. Reported uncertainties that appear unreasonably low or suspiciously high will be flagged. Participants with flagged uncertainties, particularly if they are numerous, should review their methods and ensure that the uncertainties are appropriate.

VIII. PERFORMANCE REPORTS

Participants will receive e-mail when their respective performance reports are available for review. The participant can also indicate other addresses, such as sample management offices, field offices, contracting laboratories, etc., where copies of the report should be sent. The report will include the RESL reference value for the analyte of interest, the laboratory reported value, acceptance status, and the grand mean for all laboratories. Other pertinent or helpful information may also be included. Other information about MAPEP is available on the Internet at http://www.inel.gov/resl/mapep.

MAPEP participants will not be scored or ranked. Performance, however, will be monitored and corrective actions may be called for as required. MAPEP data will also be forwarded to the Integrated Performance Evaluation Program (IPEP). IPEP will review the overall performance of the laboratory in concert with other performance evaluation programs and identify any additional concerns. Corrective actions will strive to be more focused on technical assistance rather than punitive. They may range from simple correspondence to an on-site review.

Appendix A. List of MAPEP Target Analytes and QC Parameters

Stable Inorganic Analytes

Aluminum	Antimony	Arsenic
Barium	Beryllium	Cadmium
Calcium	Chromium	Cobalt
Copper	Iron	Lead
Magnesium	Manganese	Mercury
Molybdenum	Nickel	Potassium
Selenium	Silver	Sodium
Thallium	Uranium-Total	Uranium-233
Uranium-234	Uranium-235	Uranium-238
Vanadium	Zinc	

Radiochemical Analytes

Actinium-228	Americium-241	Antimony-124
Antimony-125	Barium-133	Bismuth-212
Bismuth-214	Cadmium-109	Carbon-14
Cerium-139	Cerium-144	Cesium-134
Cesium-137	Cobalt-57	Cobalt-58
Cobalt-60	Curium-244	Europium-152
Europium-154	Europium-155	Hydrogen-3
Iodine-129	Iron-55	Lead-212
Lead-214	Manganese-54	Neptunium-237
Nickel-63	Plutonium-238	Plutonium-239/240
Polonium-210	Potassium-40	Protactinium-234m
Radium-226	Radium-228	Ruthenium-106
Selenium-75	Silver-110m	Strontium-90
Sulfur-35	Technetium-99	Thallium-208
Thorium-227	Thorium-228	Thorium-230
Thorium-232	Tin-113	Uranium-234/233
Uranium-235	Uranium-238	Yttrium-88
Zinc-65	Zirconium-95	

Semivolatile Organic Analytes

Analine	Phenol	2-Chlorophenol
1,3-Dichlorobenzene	1,4-Dichlorobenzene	Benzyl Alcohol
1,2-Dichlorobenzene	Hexachloroethane	Nitrobenzene
Isophorone	2-Nitrophenol	2,4-Dimethylphenol
2,4-Dichlorophenol	1,2,4-Trichlorobenzene	Naphthalene
Hexachlorobutadiene	4-Chloro-3-methylphenol	2-Methylnaphthalene
3 Methyl & 4-Methylphenol	Hexachlorocyclopentadiene	2-Methylphenol
2,4,6-Trichlorophenol	2,6-Dichlorophenol	o-Toluidine
1,4-Phenylenediamine	2-Chloronaphthalene	2-Nitroanaline
Dimethylphthalate	Acenaphthylene	2,6-Dinitrotoluene
3-Nitroanaline	Acenaphthene	2,4-Dinitrotoluene
2,4-Dinitrophenol	4-Chloroanaline	Dibenzofuran
4-Nitrophenol	2-Naphthylamine	1,4-Naphthoquinone
4,6-Dinitro-2-methylphenol	Diethylphthalate	Fluorene
1,2,4,5-Tetrachlorobenzene	2,4,5-Trichlorophenol	Hexachlorobenzene
Pentachlorophenol	4-Nitroanaline	Phenanthrene
Anthracene	1,4-Dinitrobenzene	1,3-Dinitrobenzene
1,2-Dinitrobenzene	Pentachlorobenzene	Pentachloronitrobenzene
Di-n-butylphthalate	2,3,4,6-Tetrachlorophenol	Fluoranthene
Pyrene	Dinoseb	Butylbenzylphthalate
Benzo(a)anthracene	Bis(2-ethylhexyl)phthalate	Chrysene
Di-n-octylphthalate	Benzo(b)fluoranthene	Benzo(k)fluoranthene
Benzo(a)pyrene	Indeno(1,2,3-c,d)pyrene	Dibenzo(a,h)anthracene
Benzo(g,h,i)perylene		

Volatile Organic Analytes

Cumene	1,1-Dichloroethane	Methylene Chloride
1,2,4-trimethylbenzene	Chloroform	1,1,1-Trichloroethane
Carbon Tetrachloride	Benzene	1,2-Dichloroethene
Trichloroethene	1,2-Dichloropropane	Bromodichloromethane
t-butylbenzene	cis-1,3-Dichloropropene	Toluene
trans-1,3-Dichloropropene	1,1,2-Trichloroethane	1,3-Dichloropropane
Tetrachloroethene	n-butylbenzene	Ethylbenzene
m & p-Xylene	1,1,2,2-Tetrachloroethane	o-Xylene

Organochlorine Pesticides

alpha-BHC	beta-BHC	gamma-BHC (Lindane)
delta-BHC	Heptachlor	Aldrin
Heptachlor epoxide	Endosulfan I	4,4'-DDE
Dieldrin	Endrin	4,4'-DDD
Endosulfan II	4,4'-DDT	Endrin Aldehyde
Endosulfan sulfate	Endrin Ketone	Methoxychlor

Appendix B. Example of a typical sample description.

RADIOLOGICAL AND INORGANIC SAMPLE DESCRIPTION
MAPEP-97-W5

The sample is a solution of 5 percent (v/v) of nitric acid in water containing some the constituents listed on the following table at the given concentrations. The sample does not contain any undissolved material. The sample also contains 25 mg/L of stable cesium added as hold-back carrier and other stable inorganic elements in a natural water matrix. The ^{234}U and ^{238}U isotopes may NOT be in equilibrium. The reference date for the radionuclides is September 1, 1997, 12:00 MST. Sample holding time is based upon the date of **RECEIPT** of the sample by the participating laboratory. Not all inorganic analytes are present in this PE sample above the lower range stated.

Analyte	Concentration Range	Analyte	Concentration Range
Sb, Ba, Be, Cr(Total), Ni, Tl, V, Zn	0.2 - 10.0 mg/L	As, Pb, Ag	0.2 - 4.0 mg/L
Cd, Se	0.2 - 0.9 mg/L	^{241}Am, ^{238}Pu, ^{239}Pu, ^{234}U, ^{238}U	< 10 Bq/L
^{90}Sr	10 - 100 Bq/L	^{57}Co, ^{137}Cs, ^{54}Mn ^{65}Zn, ^{60}Co, ^{55}Fe, ^{63}Ni	100 - 1000 Bq/L

SEMI-VOLATILE ORGANIC SAMPLE DESCRIPTION

The sample is a solution of stable inorganic, radiochemical and semivolatile organic compounds in water. This sample is to be analyzed for target semivolatile compounds only, but contains other constituents to simulate the radiological/inorganic sample. The sample contains the constituents listed on the following table at the given concentrations. The sample should not contain any undissolved material.

Analyte Class	Concentration Range	Analyte Class	Concentration Range
Phthalate Esters	<100 micrograms/Liter	Polynuclear Aromatics	<100 micrograms/Liter
Phenols	<300 micrograms/Liter	Nitroaromatics	<200 micrograms/Liter
Chlorinated Hydorcarbons	<100 micrograms/Liter		

"MAPEP samples are analytical standards or a "product" generated for the purpose of securing and evaluating analytical services; they are not hazardous waste and they are not samples of hazardous waste... Thus, a laboratory participating in the MAPEP is in the process of establishing its eligibility and credentials to do DOE analytical work. It follows, therefore, that the laboratory is the "generator" of the waste resulting when the samples and the resulting residues are to be discarded." (MEMORANDUM OCC-95-189, Office of Chief Counsel, October 16, 1995)

Appendix C. Method codes for radionuclides.

RADIONUCLIDES

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1. The first pair of digits designates the method of detection (instrument).

00 Alpha Spectrometry
01 Beta Counting - gas flow proportional counter
02 Beta Counting - liquid scintillation counter
03 Gamma Spectrometry
04 Gross Alpha/Beta - gas flow proportional counter
05 Thermal Ionization Mass Spectrometry
06 Photon Electron Rejecting Alpha Liquid Scintillation
07 Kinetic Phosphorescence Analyzer (KPA)
08 Inductively Coupled Plasma Mass Spectrometer (ICP-MS)
99 Other

*
σσ

2. The second pair of digits designates sample treatment by the laboratory upon receipt.

00 No pretreatment - analyzed as received
01 Filtration
02 Centrifugation
03 Drying
04 Grinding, sieving, or blending , no drying
05 Grinding, sieving, or blending and drying
99 Other

*
σσ

3. The third pair of digits designates the sample preparation technique.

00 No preparation - analyzed as received
01 Evaporation, straight
02 Evaporation, acidified
03 Coprecipitation, straight
04 Coprecipitation, acidified
05 Distillation
06 Acid leaching
07 Acid digestion - the use of oxidizers to destroy organics
08 Acid dissolution by strong Aqua Regia, hydrofluoric acid, etc.
09 Total dissolution by fusion
99 Other

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4. The * is a letter (A through G) indicating sample size.

Appendix D. Method codes for inorganic metals (see Appendix C for code positioning).

INORGANIC METALS

1. The first pair of digits designates the method of detection (instrument).

00 Flame Atomic Absorption Spectrometry
01 Furnace Atomic Absorption Spectrometry (Zeeman Background Correction)
02 Radial-Inductively Coupled Plasma Emission Spectrometry
03 Axial-Inductively Coupled Plasma Emission Spectrometry
04 Inductively Coupled Plasma Mass Spectrometry
05 Cold Vapor Atomic Absorption Spectrometry
06 Hydride Generation (AAS, ICP/OES, ICP-MS)
07 DC Plasma Emission
08 Furnace Atomic Absorption Spectrometry (Deuterium Continuum Background)
09 Ion Chromatography - EPA Method
10 Flame Emission Spectrometry
11 Thermal Ionization Mass Spectrometry
99 Other

2. The second pair of digits designates sample treatment by the laboratory upon receipt.

00 No pretreatment - analyzed as received
01 Filtration
02 Centrifugation
03 Drying of sample prior to sample preparation
04 Grinding, sieving, blending and no drying
05 Grinding, sieving, blending and drying
99 Other

3. The third pair of digits designates the sample preparation technique.

00 No preparation - analyzed as received
01 Hot acid leaching - EPA Methods 3010, 3020, 3050 or CLP ILM03.0
02 Microwave assisted - EPA Methods 3015, 3051, or CLP ILM03.0
03 Concentrated nitric acid and/or other oxidizers to destroy organics
04 Acid dissolution by strong Aqua Regia, hydrofluoric acid, etc.
05 Total dissolution by fusion
99 Other

4. The * is a letter (A through G) indicating sample size.

Appendix E. Method codes for organic analytes (see Appendix C for code positioning).

ORGANIC ANALYTES

1. The first pair of digits designates the method of analysis.

00 USEPA Method 601 - Purgable Halocarbons
01 USEPA Method 602 - Purgable Aromatics
02 USEPA Method 608 - Organochlorine Pesticides and PCB's
03 USEPA Method 624 - Purgables
04 USEPA Method 625 - Base/Neutrals and Acids
05 SW-846 8240B Volatile Organics
06 SW-846 8260 Volatile Organic Compounds
07 SW-846 8270 Semivolatile Organic Compounds
08 SW-846 8081A Organochlorine Pesticides by Gas Chromatography
09 SW-846 8082 Polychlorinated Biphenyls by Gas Chromatography
10 SW-846 8091 Nitroaromatics and Cyclic Ketones by Gas Chromatography
11 SW-846 8100 Polynuclear Aromatic Hydrocarbons
12 SW-846 8121 Chlorinated Hydrocarbons by Gas Chromatography: Capillary
13 SW-846 8260B Volatile Organics Compounds by GC/MS
14 SW-846 8270C Semivolatile Organic Compounds by GC/MS
15 SW-846 8275A Semivolatile Organic Compounds (PAHs and PCBs) TE/GC/MS
16 SW-846 8310 Polynuclear Aromatic Hydrocarbons
17 SW-846 GC/GTIR for Semivolatile Organics: Capillary Column
99 Other

2. The second pair of digits designates PRIMARY sample cleanup methodologies.

00 No cleanup
01 Gel Permeation Chromatography Cleanup
02 Adsorption Column Chromatography (Silica Gel, Alumina, Florisil)
03 Acid-base partioning
04 Sulfur cleanup
99 Other

(continued on next page)

3. The third pair of digits designates the sample preparation technique/method.

00 No Preparation
01 Separatory Funnel Liquid-Liquid Extraction
02 Continuous Liquid-Liquid Extraction
03 Soxhlet Extraction (Method 3540)
04 Automated Soxhlet Extraction (Method 3541)
05 Pressurized Fluid Extraction (Method 3560)
06 Ultrasonic Extraction (Method 3550B)
07 Supercritical Fluid Extraction of PAHs (Method 3561)
08 Waste Dilution for Volatile Organics (Method 3585)
09 Purge-and-Trap for Aqueous Samples (Method 5030B)
10 Closed-System-Purge-and-Trap and Extraction for Volatiles (Method 5035)
99 Other

4. The * is a letter (A through G) indicating sample size.

For All Analyte Types, the '*' corresponds to values in the following table.

Sample Size Table

A less than 1 gram or 1 milliliter
B 1 to 5 grams or 1 to 5 milliliters
C 6 to 10 grams or 6 to 10 milliliters
D 11 to 30 grams or 11 to 30 milliliters
E 31 to 75 grams or 31 to 75 milliliters
F 76 to 100 grams or 76 to 100 milliliters
G 101+ grams or 101+ milliliters

Appendix F

MIXED ANALYTE PERFORMANCE EVALUATION PROGRAM (MAPEP)
DATA ENTRY INSTRUCTIONS (05/04)

PRELIMINARY CONSIDERATIONS:

Due to the multiplicity of potential Internet web browsers, products other than Microsoft's Internet Explorer or Netscape may operate the reporting software as well with no issues. Laboratory personnel using other products may have to try out their browser with the reporting software to ascertain if any issues arise.

While MAPEP is awaiting all laboratory data to be entered, the MAPEP system is read/write. After the MAPEP closing date, the reporting system becomes READ ONLY so users can ONLY review the data they have entered into the system or review previous MAPEP studies. When a new MAPEP standard is distributed, the MAPEP system will once again be ready for data entry for the new sample.

DATA ENTRY AND/OR EDITING:

1) Start you computer's Web Browser software.
Type in the URL http://mapep.inel.gov/

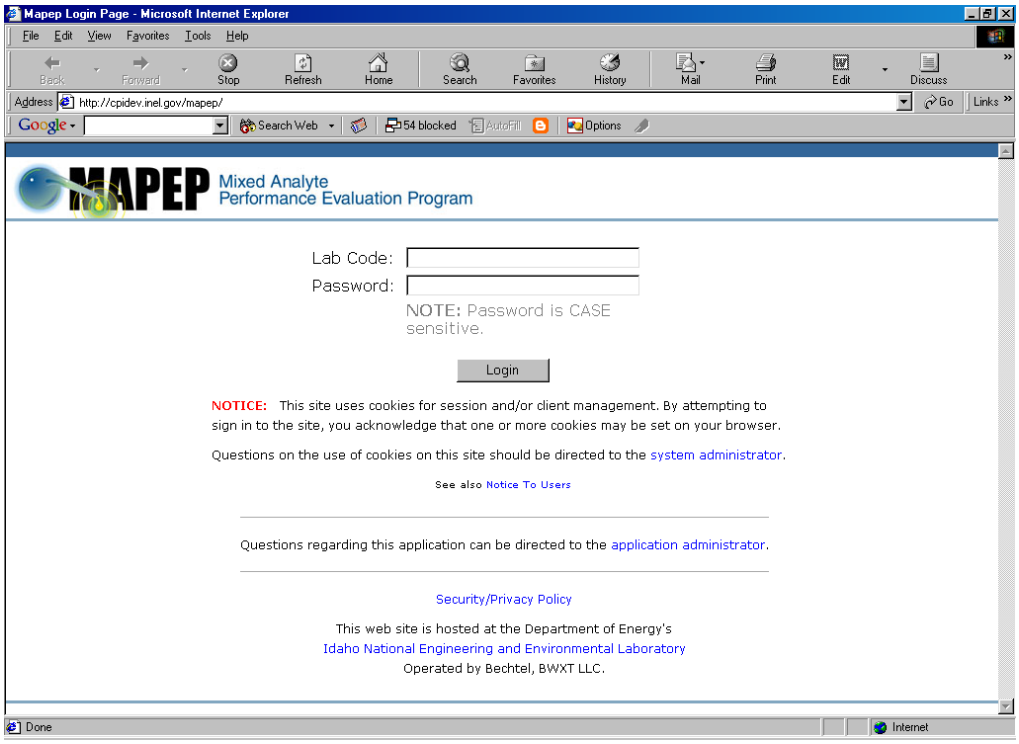
WARNING: You must ***LOG OFF*** the data entry program. Simply closing your browser will not log you off the MAPEP server and additional attempts to LOG IN will fail until the system resets itself (approximately 20 minutes).

ALSO: The SUBMIT buttons may be at the bottom of some user screen depending upon your individual terminal resolution. Users with lower screen resolution will have to scroll down to get to these buttons.

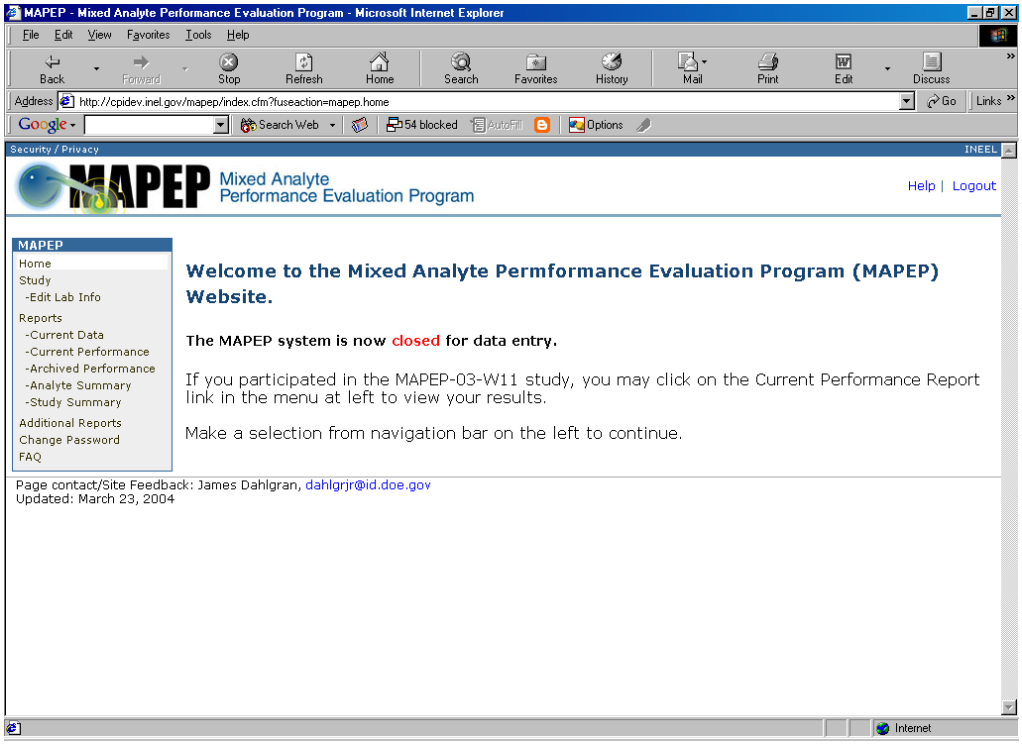
The Following Welcome screen appears:

1) Enter your Lab Code and Supplied password and then Click on the Login Button.

NOTE: Laboratories now have control of their passwords and change them as they desire, HOWEVER they must meet certain security criteria. See page 25



2) The Welcome Screen is displayed that will tell you the status of each of the MAPEP studies currently distributed or being evaluated.

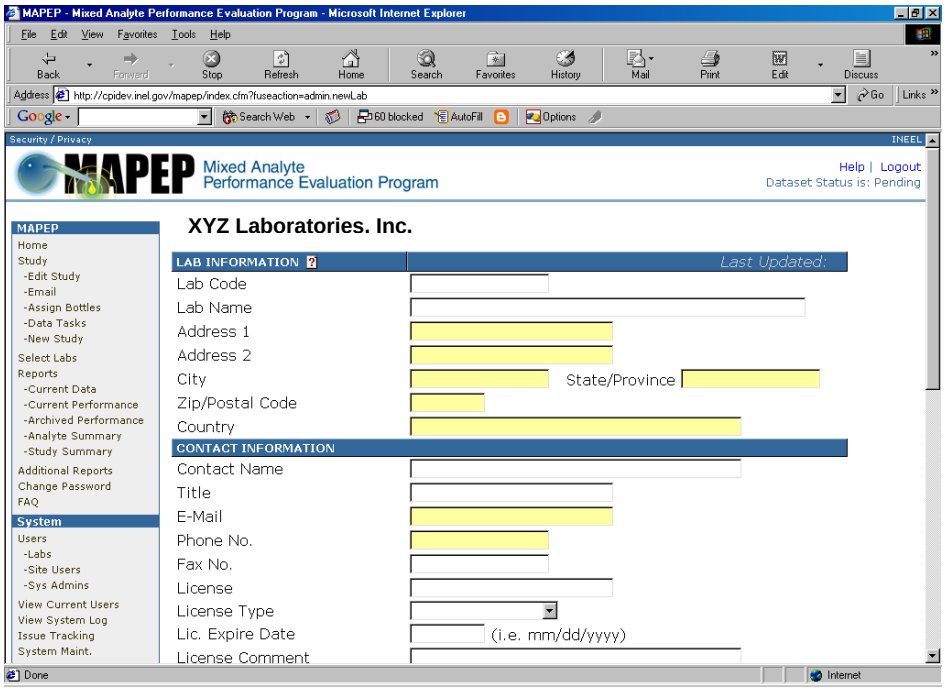


3) Users are REQUIRED to maintain the Laboratory Information up to date as this is the primary way MAPEP will use to communicate with the participants.

For each new study, the MAPEP users MUST validate the laboratory information before they are allowed to enter data.

To change data in a cell, click in that cell.

DO NOT ENTER POST OFFICE BOX INFORMATION IN THE SHIPPING INFORMATION AREA.



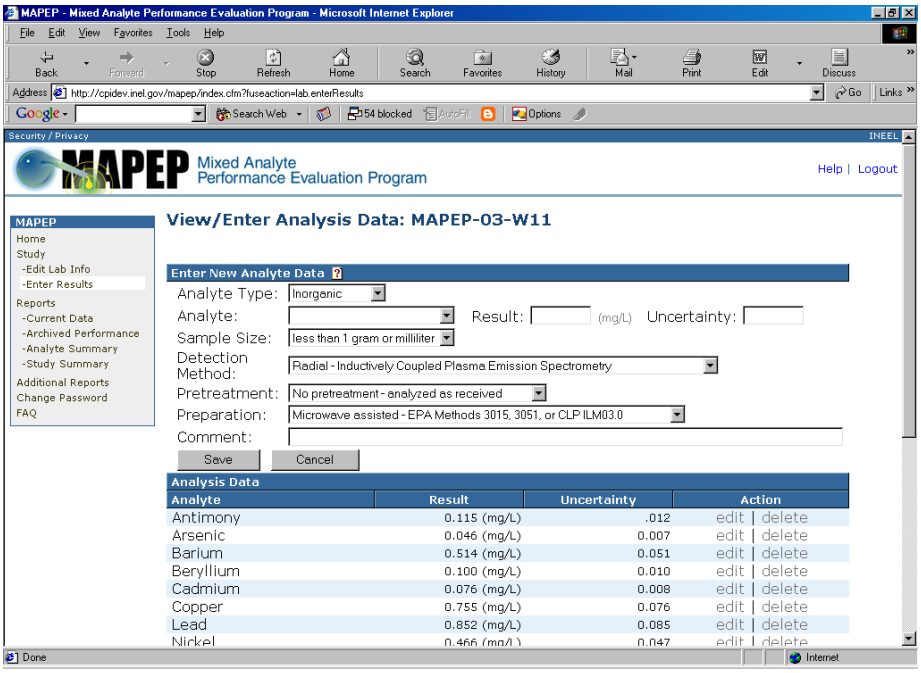
The participant's NRC license or state license number, and the expiration date, must be provided for ALL United States Laboratories. If a license exemption applies, the user must enter the appropriate DOE contract number and expiration date. A U.S. Federal Laboratory (owned and operated by the federal government) may enter any appropriate license information or select the federal laboratory option. A foreign laboratory (outside U.S. jurisdiction) will not see the NRC License request, as this option does not apply.

When users get to the shipping information, they may elect to check the "Same as Mailing Info" and/or "Same as Contact Info" to help provide information for shipping.

Once the user has updated their laboratory information, at the **bottom** of the screen click the **SAVE** button.

4) If your facility requests your MAPEP reports to be sent to other facilities, you may specify these facilities by clicking on ADDITIONAL REPORTS link in the menu window on the left of the screen display.

Users may now enter their analytical data:



5) As long as the data session is open, you may click on ENTER RESULTS on the menu bar to input or edit your results. When the data session has closed, this menu option will disappear.

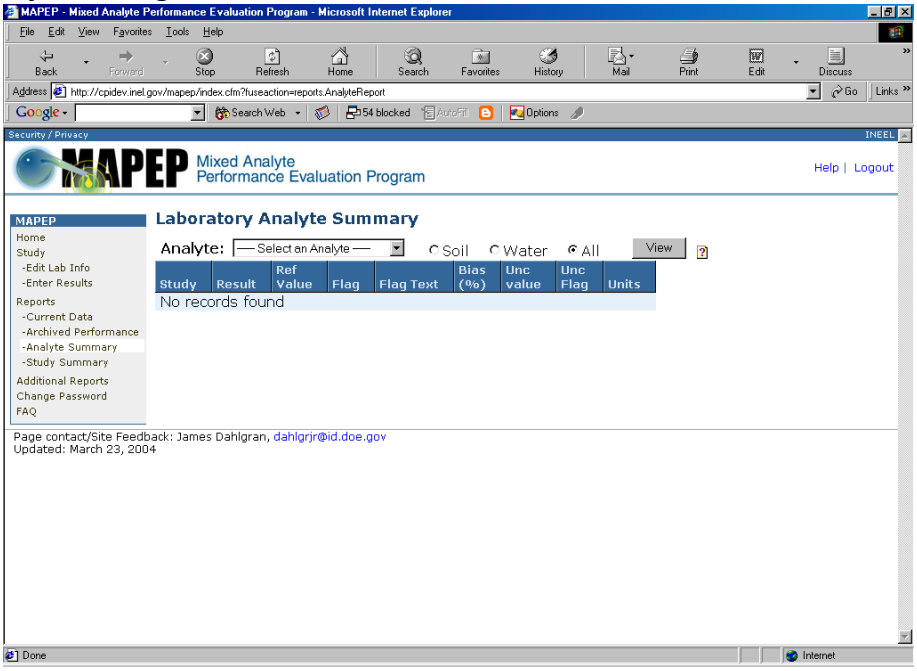
Select the appropriate analyte type (radiological, inorganic, volatile, semivolatile or pesticides) to start reporting data. The appropriate analyte list, units, and potential method codes are presented based upon the analyte type selected. The Web reporting system for MAPEP is very similar to the previous version.

After each data point has been entered, the user must click the SAVE button at the bottom of the data entry area to save the data. The list of data entered appears below the data entry area.

You will notice that to the far right of each of the analytes entered is a EDIT | delete action button. This allows users to edit the data entered for the analyte chosen or you may delete that analyte as necessary.

You will also notice that if you hoover the mouse pointer over the name of the analyte, a small pop-up window appears that gives you details of the data you have entered.

6) From the data entry screen, you may elect to go the REPORTS section of the Website that will allow the user to view and/or printout reports of the currently entered data (CURRENT DATA) or historical data (ARCHIVED PERFORMANCE) reported under previous studies.

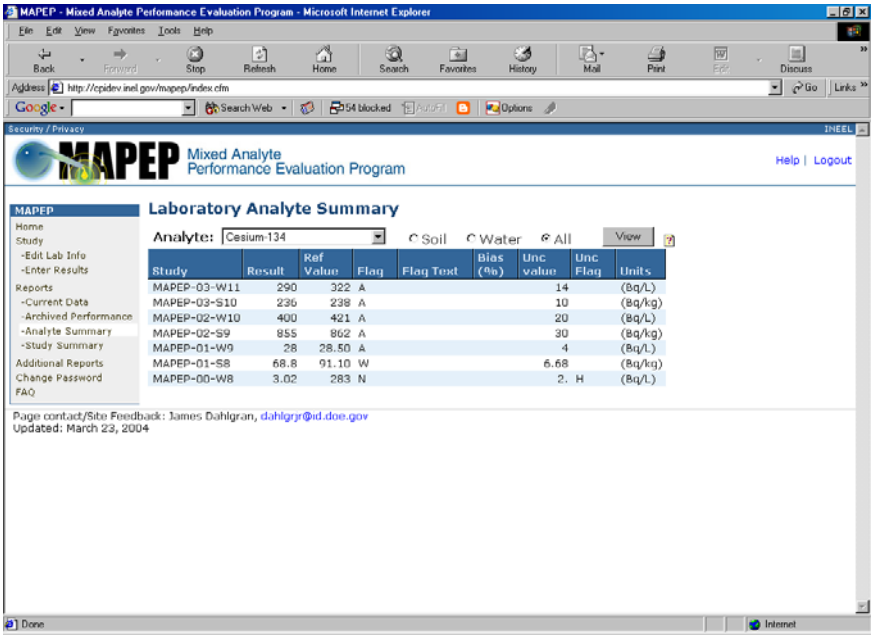


7) With this version of the MAPEP Website, users can ascertain their historical performance for any analyte they have reported earlier.

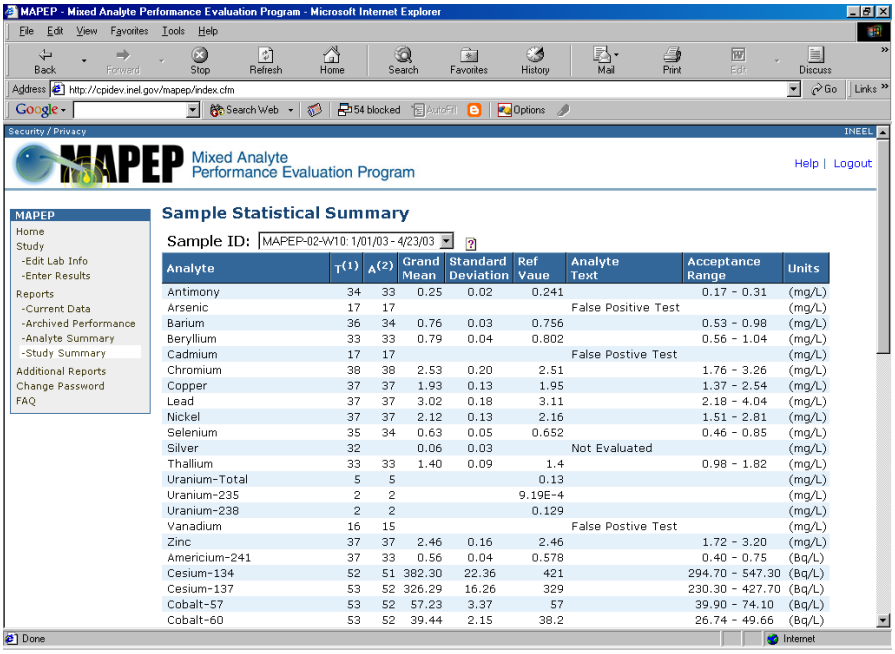
Click on the menu item ANALYTE SUMMARY to access this function.

From the dropdown menu window, select an analyte you wish to review. Then select

whether you wish to review this performance in water, soil or all. Finally, click the VIEW button to retrieve the analyte specific performance data.



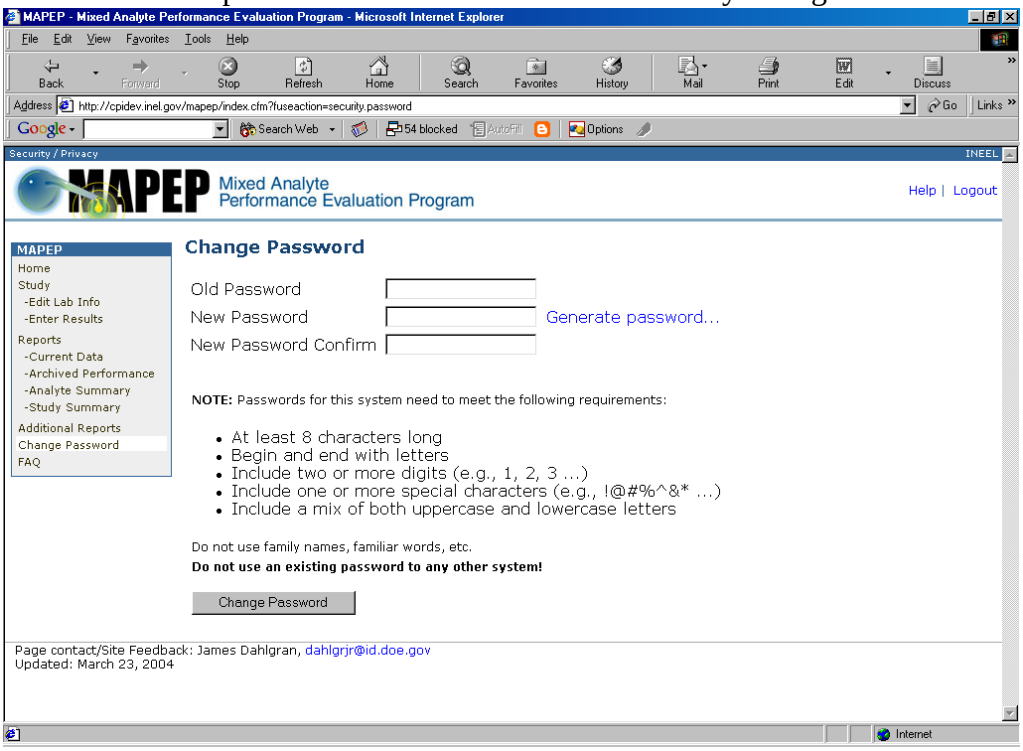
An additional report STUDY SUMMARY allows users to review the historical performance of past studies. Click on this menu item to generate the report like that to the right.



The MAPEP reporting system allows users to control their own passwords (within limits). Passwords must be changed or updated every six months. Passwords must meet the following criteria for security reasons.

- At least 8 characters long
- Begin and end with letters
- Include two or more digits (e.g., 1, 2, 3 ...)
- Include one or more special characters (e.g., !@#%^&* ...)
- Include a mix of both uppercase and lowercase letters

To change your password, click the CHANGE PASSWORD menu item and the screen will appear. There is a Generate Password tool incorporated into this screen that will allow you to generate a compliant password if you desire. Just click on this link and a pop-up window will appear with a suggested password.



DATA MODIFICATION OR DELETION

1) If it is desirable to modify or delete data entries from the data entered, Click on the "ENTER RESULTS" menu item while the study is open. The list of analytes entered will appear below the data entry area. To the far right of each of the analytes you will notice the "edit | delete" selection. Selecting the edit function will allow you to edit the data entered for this analyte. Selecting DELETE will delete the analyte from the list of analytes reported and from the database.

LOG OFF

1) To exit the MAPEP data entry program, select LOG OFF from upper right menu bar. Your data and information will be saved for your update and/or review at anytime.

DO NOT CLOSE YOUR BROWSER PROGRAM (WINDOW) UNTIL YOU HAVE LOGGED OFF. THIS WILL LOCK YOU OUT OF ADDITIONAL SESSIONS FOR 20 MINUTES UNTIL THE SERVER RESETS.

Keep the password, instructions, and any hard copy in a secure location. If you have problems or questions, please call Guy M. Marlette at (208) 526-2532 or e-mail us MAPEP@INEL.GOV.